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Assembly of transition metal ion on cellulose surface anchored with azo-Schiff base and its catalytic activity for H_2O_2 decomposition

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ABSTRACT

To form polymer-supported catalysts, to eliminate excess H_2O_2 during wastewater treatment, the assembly of metal ion on cellulose was investigated. Cellulose was covalently grafted with reactive azo-Schiff base containing azo-conjugated system, 3,5-bis {2-hydroxyphenyl-5-[(2-sulfate-4-sulfatoethylsulphonyl-azobenzol) methylene amino]} benzoic acid. Metal ions, Co^{2+}, Mn^{2+}, and Cu^{2+}, were anchored to the modified cellulose by controlling assembly. The cellulose-supported complexes were characterized and the catalytic activity for H_2O_2 was investigated. Elemental analyses of the cellulose supported complexes, MC–Co, MC–Mn, were well agreed with the proposed composition. Three cellulose-supported complexes had strong catalytic activities for H_2O_2. H_2O_2 decomposition rates in 10 min were 84.54% for MC–Co, 94.87%, for MC–Mn, and 77.21% for MC–Cu, respectively. The manganese complex had the strongest catalytic activity among them. The cellulose-supported complexes have potential application in wastewater treatment and biotechnology.

Keywords: Cellulose; Catalytic supported; Schiff base; Complexes; H_2O_2 decomposition

1. Introduction

In the last years, polymer-supported catalysts for wastewater treatment have received considerable interest among the academia and industries due to their unique structures and excellent properties [1–3]. The polymer-supported catalysts enhance their thermal stability, selectivity, recyclability, and easy separation from wastewater or reaction products [4]. H_2O_2, as cleaner oxidation reagent, has been widely applied in environmental treatment, textile industry, food industry, and other industrial areas [5–7]. For example, Fenton's reagent combined with H_2O_2 represents one of the most popular advanced oxidation processes which allow degradation of organic pollutants and dyestuffs in water into carbon dioxide. H_2O_2 is also used in large-scale municipal water treatments. Moreover, hydrogen peroxide has been applied to cold pasteurization of milk to prevent microbial pollution in the food chemistry. It is an important topic to eliminate excess H_2O_2 in wastewater treatment after the oxidation reaction is finished [8,9].

Some transition metal ions, such as Co^{2+}, Mn^{2+}, Fe^{2+}, and their complexes, not only catalyze hydrocarbon oxidation by molecular oxygen or various oxygen donors, but also catalyze hydrogen peroxide decomposition [9–12]. It is very difficult to separate and remove the transition metal ions and their

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complexes from water. This can result in the second water contamination. So, the immobilization of homogeneous catalysts onto polymer supports through covalent attachment has received attention because these materials have advantageous feature of heterogeneous catalysts to homogeneous systems [13–15]. The supported catalysts can be easily recovered without any substantial loss during their catalytic activity. Polymer supports have been found to be more useful as they are able to provide preferential stereochemistry for catalysts and reactions [4,14,16,17].

Cellulose is one of the natural biomaterials, which is an excellent catalyst-supported materials [13,18,19]. Cellulose fiber has been explored as a substrate for composite materials because of the presence of functional groups that may be employed in various activation processes. A lot of attempts have been carried out to make cellulose-based nanocomposites. These composite materials are biocompatible, biodegradable, and possess low toxicity [20–22]. In our previous research work [21,23], some Schiff base compounds, such as N,N-bis{p-[(2´-sulfatoethyl)sulfonyl phenylazo] salicylidene}-1,2-ethylenediamine (BSPEA), and 3,5-bis{2-hydroxyphenyl-5-[(2-sulfate-4-sulpftoethylsulfonyl-azobenzol) methylene amino]} benzoic acid (BHSABA), were applied to modify cellulose. The modified cellulose fabrics had excellent UV-protective property and good antibacterial activity against *S. aureus*. The multi-functional cellulose will have potential application in biomaterials and hi-tech textile fields. Schiff base ligands are able to coordinate metal through imine nitrogen and another group [24,25]. The Schiff base complexes of transition metals are efficient in loading of oxygen and mimicking the enzyme activity, which play a significant role in many catalytic reactions like oxidations, asymmetric cyclopropanation, and polymerization [14,25–27]. 3,5-di-aminobenzoic acid, as an important intermediate, is applied to synthesize some Schiff base compounds. Schiff base with 3,5-di-aminobenzoic acid has excellent coordination property with transition metal ions [22,28]. However, the reports of transition metal Schiff base complexes on the supported cellulose are scarce. The modification of cellulose with Schiff base needs the compounds containing reactive groups. Transition metal ions can be controlled to assemble on the grafted cellulose with suitable Schiff base.

In this paper, to eliminate excess H_2O_2 during wastewater treatment, a new method was investigated. A novel reactive ligand containing azo-conjugated system, 3,5-bis {2-hydroxyphenyl-5-[(2-sulfate-4-sulfatoethylsulfonyl-azobenzol) methylene amino]} benzoic acid, was applied to modify cellulose surface. The modified cellulose surface was controlled to assemble with transition metal ion. The cellulose-supported complexes were characterized by elemental analysis, FT-IR, ^{1}HNMR, TG-DTG, scanning electron microscopy (SEM), and energy disperse spectroscopy (EDS) methods. Their catalytic activities for H_2O_2 decomposition were also investigated.

2. Experimental

2.1. Materials

Desized, scoured, and bleached cellulose fabric was obtained from Jinqiu Textile and Finishing Company, Shaoxing, China. 3,5-bis {2-hydroxyphenyl-5-[(2-sulfate-4-sulfatoethylsulfonyl-azobenzol) methylene amino]} benzoic acid (BHSABA) Schiff base was obtained from National Engineering Research Center for Dyeing and Finishing of Textiles, Shanghai, China. Chemical structure of BHSABA is shown in Fig. 1.

Metal salts, $Mn(Ac)_2{\cdot}4H_2O$, $Co(Ac)_2{\cdot}4H_2O$, $Cu(Ac)_2{\cdot}H_2O$, and other chemicals used were purchased from Shanghai Chemical Reagent Plant, Shanghai, China.

2.2. Grafting of cellulose fabric with BHSABA Schiff base

Grafting reaction of cellulose fabric with BHSABA Schiff base was carried out in the similar way as described in the reference [21]. The concentration of BHSABA Schiff base was 10% (Schiff base/cellulose fabric, w/w). Sodium carbonate (10 g/l), as catalyst, was used in the grafting process. Fabric was immersed in BHSABA solution at room temperature and the temperature was increased to 60℃. The grafting was carried out at 60℃ for 50 min. The modified samples were rinsed in hot water and soaped in a solution of a nonionic surfactant (OP-10, 1 g/l) at 85℃ for 15 min at liquor ratio 1:15. The samples were removed, rinsed thoroughly in hot tap water, and then dried at the ambient condition.

Grafting reactive rate (R) was calculated according to Eq. (1) by measuring the absorbance of the residual BHSABA Schiff base at 402 nm. Grafting quantity (G) of BHSABA Schiff base on the cellulose fabric was calculated according to Eq. (2):

$$R\,(\%) = [(A_0 - A_1 - A_2)/A_0] \times 100\% \tag{1}$$

$$G\,(\text{mmol/g}) = C \times R/MW \tag{2}$$

where A_0 and A_1 are the absorbance of the Schiff base solution at $\lambda_{\max}$ before and after grafting, respectively; A_2 is the absorbance of the soaped Schiff base solution

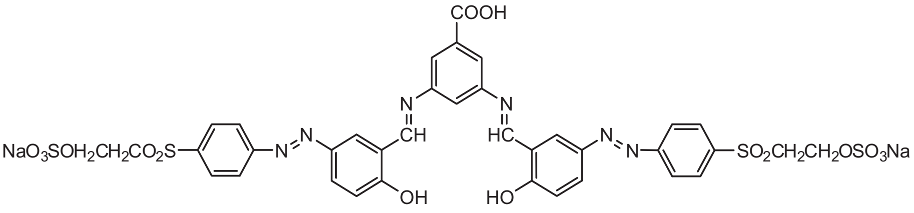

Fig. 1. Chemical structure of BHSABA Schiff base.

with a nonionic surfactant (OP-10, 1 g/l). *C* is the weight concentration of BHSABA Schiff base. *W* is the weight of the grafted cellulose. *M* is the molecular weight of BHSABA ($M = 944$). The grafted cellulose sample was named as MC (modified cellulose).

2.3. Assembly of transition metal ions on cellulose

The grafted cellulose sample, 1 g, was immersed in the absolute EtOH, 30 ml. The certain quantity of metal salts (1:2 mol ratio of Schiff base ligand to the metal ion), $Mn(Ac)_2{\cdot}4H_2O$, $Co(Ac)_2{\cdot}4H_2O$ and $Cu(Ac)_2{\cdot}H_2O$, was dissolved in absolute EtOH, 20 ml, and added to the EtOH solution containing grafted cellulose, respectively. The mixture was heated to boil and reflux for 6 h. After the assemble reaction was completed, the samples were taken out and rinsed in hot water, and soaped in a solution of a nonionic surfactant (OP-10, 1 g/l) at 85˚C for 15 min at liquor ratio 1:15. The samples were removed, rinsed thoroughly in hot tap water. Then, the samples were dried at the ambient condition. The modified samples containing assembly transition metal ions were named as MC–Co, MC–Mn, and MC–Co, respectively.

2.4. Characterization of the cellulose-supported complexes

Element analysis was measured with Elemental Analyzer, Vario ELIII (Elmentar, Germany). The samples were dried under vacuum at the temperature of 50˚C before measuring. Metal element content of the complexes was measured by Inductively Coupled Plasma-Atomic Emission Spectrometer, Prodigy (Leeman, USA). FT-IR spectrum of the sample was measured by an OMNI Sampler of the Nexus-670 FT-IR-Raman Spectrometer (Nicolet Analytical Instruments, Madison WI).

For SEM and EDS analysis, the samples were sputtered with gold and then examined with a IE 300 X (Oxford, UK).

Thermogravimetric analysis (TG) was carried out at a heating rate of 10˚C/min in nitrogen using a Thermogravimetric Analyzer, TG 209 F1 (NETZSCH-Geraetebau GmbH, Germany).

2.5. Measurement of catalytic activity for H_2O_2 decomposition

The modified cellulose sample, 0.5 g, was added to H_2O_2 solution with magnetic stirring. An initial concentration of H_2O_2 was 10 g/l and NaOH being 1 g/l. The temperature of catalytic reaction for H_2O_2 decomposition was set to 40˚C. Once the reaction was started, the reaction media, 1 ml, was added to the H_2SO_4 solution (98% H_2SO_4:water 1:5, v/v), 3 ml, at different reaction time. The concentration of residual H_2O_2 was measured by means of a titration with 0.02 mol/l $KMnO_4$. The rate of H_2O_2 decomposition (*D*) is calculated by Eq. (3):

$$D\,(\%) = [(C_0 - C_1)/C_0] \times 100\% \tag{3}$$

where C_0 is the initial concentration of H_2O_2, C_1 is the concentration of residual H_2O_2 at different reacting time.

3. Results and discussion

3.1. Modification of cellulose with the azo-Schiff base

BHSABA Schiff base with azo-conjugated system contains two symmetrical reactive groups, p-[(2´-sulfatoethyl)sulfonyl] benzol group. p-[(2´-sulfatoethyl)sulfonyl] benzol group has high reactivity. There are a lot of hydroxyls on cellulose that may be employed in various activation processes. BHSABA Schiff base is able to form covalent bonds with cellulose under the alkaline condition by the crossing reaction. The chemical reaction of cellulose and BHSABA Schiff base is shown in Fig. 2.

UV–vis spectrum of BHSABA Schiff base shows that the ligand band in the 350–450 nm range had π–π^* transition of the ligand-conjugated system. The absorbance peak of ligand BHSABA Schiff base in visible

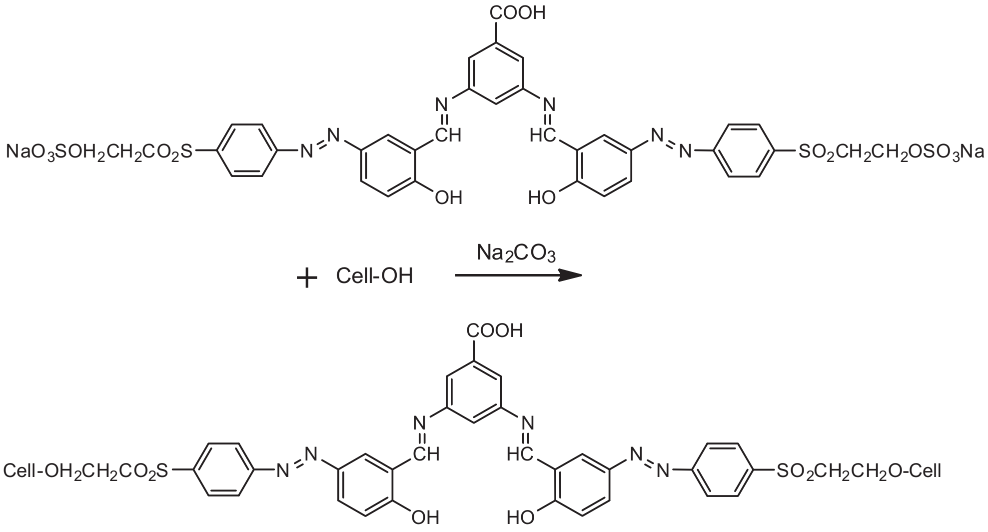

Fig. 2. Mechanism of cellulose grafted with BHSABA.

range ($\lambda_{\max}$) occurred at 402 nm. This might be attributed to π–π^* and n–π^* transition of Schiff base containing azo-conjugated system. Grafting reactive rate can be calculated by measuring the absorbance of the residual BHSABA Schiff base at 402 nm. The grafting reactive rate on cellulose was 24%. According to Eqs. (1) and (2), the grafting quantity (G) of BHSABA Schiff base on cellulose fabric was 5.08×10^{-2} mmol/g.

The element analyses of the grafted cellulose and control sample were determined. The nitrogen content of MC and the control samples was 0.035 and 0%, respectively. Compared with the control sample, the nitrogen content of MC increased. Reflectance spectra of MC and the control sample were measured and shown in Fig. 3. It indicates that the control cellulose had no absorbance at 350–700 nm. But MC had a strong absorbance at 350–500 nm, resulting from π–π^* and n–π^* transition of carbonyl and azomethine groups and the π–π^* transition of C=C bonds available in aromatic rings. It can be seen that BHSABA Schiff base compound was anchored to cellulose macromolecules by covalently grafted way.

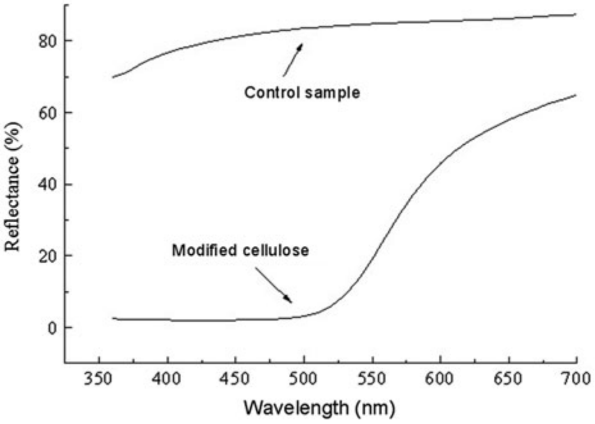

Fig. 3. Reflectance spectra of MC and control samples.

FT-IR spectrum of MC was also measured. The infrared band of –COOH is showed at 1,651 cm^{-1}. FT-IR spectrum assured the presence of –N=N– at 1,583 cm^{-1} and –N=C– at 1,514 cm^{-1}. This further confirms that the Schiff base compound was able to form covalent bonds with cellulose. The results were in agreement with the reference [23].

3.2. *Assembly of transition metal ions on cellulose surface anchored with azo-Schiff base*

Schiff base ligands are able to coordinate metal through imine nitrogen and hydroxy groups. The coordination chemistry of transition metal with diverse ligands remains an area of considerable interest [25,29,30]. Schiff base with azo-conjugated system anchored to cellulose can locate to coordinate transition metal ions and form controlling assembly of metal ion complexes on cellulose surface. The assembly reaction of metal ion complexes on cellulose surface is shown in Fig. 4.

Schiff base based on 3,5-di-aminobenzoic acid has excellent coordination property with transition metal ions. The proposed structures based on 3,5-diaminobenzoic acid supported 1:2 mol ratio of Schiff base ligand to the metal ion according to the references [21,27]. The metal element analyses of the

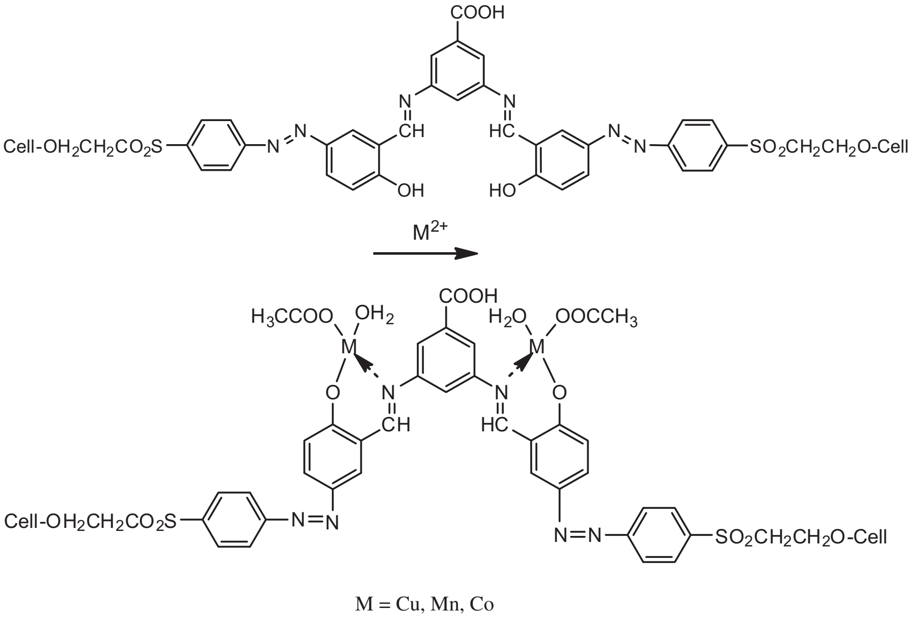

Fig. 4. Formation and proposed structures of metal ions on cellulose surface.

cellulose complexes, MC–Co, MC–Mn, and MC–Cu, were determined by Inductively Coupled Plasma-Atomic Emission Spectrometer and the theoretical results of metal were calculated according to chemical formula, and listed in Table 1. Co content for MC–Co was 0.57%, Mn content for MC–Mn was 0.61%, and Cu content for MC–Cu was 1.53%, respectively. According to the proposed structures and the theoretical calculation of the grafting quantity (*G*) of BHSABA Schiff base on cellulose fabric ($G = 5.08 \times 10^{-2}$ mmol/g), the metal elements of the cellulose complexes, MC–Co, MC–Mn, and MC–Cu, were 0.59, 0.56, and 0.65%, respectively. Elemental analysis of the cellulose-supported complexes, MC–Co and MC–Mn, were well agreed with the proposed composition. Cu content of MC–Cu was higher than that of the theoretical calculation. The naked copper ion was easily attracted on the cellulose surface and it was difficult to wash away by water. There are many hydroxyl groups on cellulose macromolecules, which can form relatively stable complexes with copper ion.

Table 1
Metal contents of the cellulose supported complexes

Samples	MC–Co (%)	MC–Mn (%)	MC–Cu (%)
Measured results	0.57	0.61	1.53
Theoretical results	0.59	0.56	0.65

Micro-FT-IR spectra of the cellulose-supported complexes were measured and shown in Fig. 5. Because of the effect of metal ion coordinate, the complex peaks of –COOH moved to 1,632 cm^{-1} for MC–Co, 1,635 cm^{-1} for MC–Mn, and 1,614 cm^{-1} for MC–Cu, respectively. There were overlapping areas between the peaks of –COOH and –C=N– (Fig. 2(a)). In the spectrum range of 600–750 cm^{-1}, there were three peaks, 618 cm^{-1} for MC–Co, 619 cm^{-1} for MC–Mn, and 621 cm^{-1} for MC–Cu, respectively. They are attributed to corresponding O-M bonds. This further confirms that the modified cellulose with azo-Schiff base can be controlled to assemble with transition metal ions.

3.3. Morphological structure of the cellulose supported complexes

The morphological structure and surface element analysis of the cellulose-supported complexes were investigated. SEM analysis was then used to characterize the changes in the surface morphology. EDS analysis was used to determine the carbon, oxygen, and metal element contents on the surface of the cellulose-supported complexes. The results are shown in Fig. 6(a)–(d). Compared with the control cellulose, the appearances of MC–Co, MC–Mn, and MC–Cu samples had no notable difference. The appearances of the metal cellulose supported complexes were smooth, which demonstrates that the assembly complexes existed in cellulose matrix in molecular way by

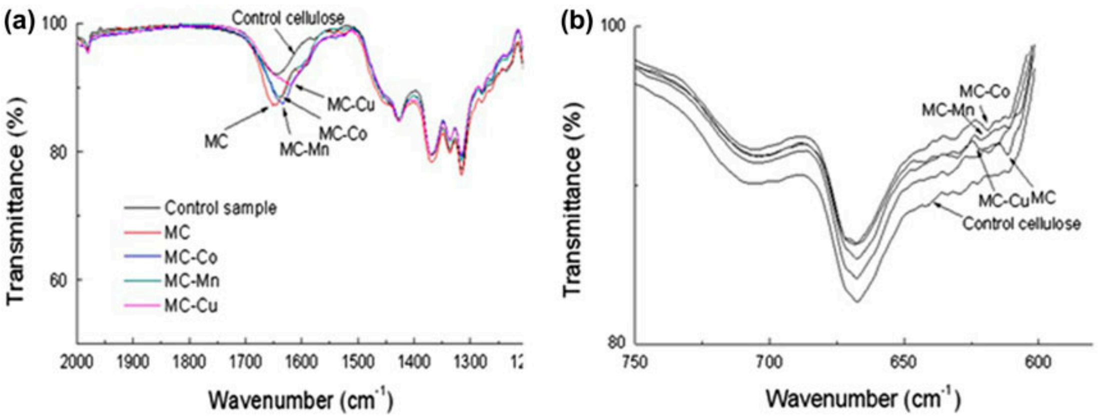

Fig. 5. FT-IR spectrum of MC and the cellulose supported complexes: (a) micro-infrared spectrum of complexes in 1200–2000 cm^{-1} and (b) micro-infrared spectrum of metal bonds.

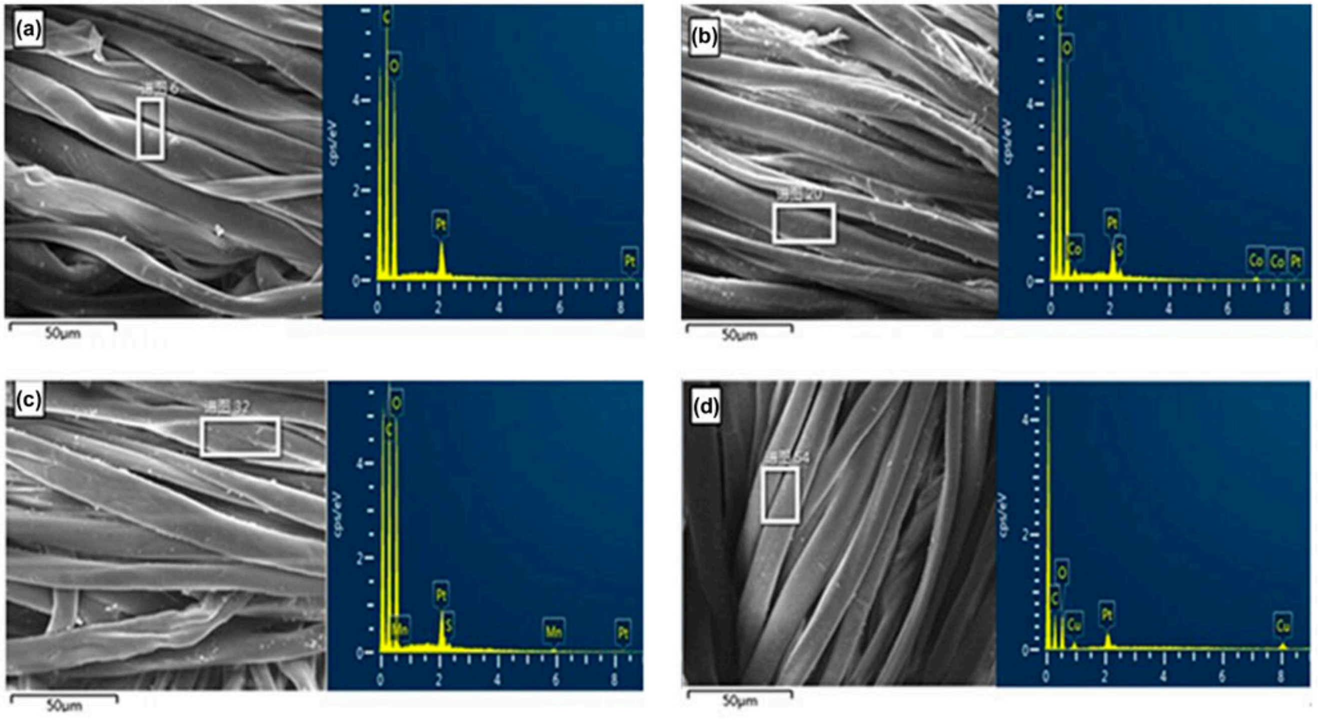

Fig. 6. SEM-EDS analysis of control cellulose and the cellulose supported complexes: (a) MC, (b) MC-Co, (c) MC-Mn, and (d) MC-Cu.

grafting method. The results of EDS analysis are listed in Table 2. The metal elements of the cellulose-supported complexes for MC–Co, MC–Mn, and MC–Cu were 0.71, 0.63, and 7.39% (wt), respectively. The values of MC–Co, MC–Mn were basically in agreement with the theoretically calculated amount and element analysis amount. The small sulfur peak in MC–Co and MC–Mn, might be due to the presence of Na_2SO_4, which was absorbed on the cellulose and were not absolutely removed by washing. The value of MC–Cu was obviously higher than that of the theoretical amount calculated and element analysis amount. Copper ion might be easily attracted and aggregated on the surface of cellulose. The results of EDS analysis represented surface chemical composition on the cellulose-supported complexes.

Table 2
Element contents on the surface of the cellulose supported complexes

	MC–Co		MC–Mn (%)		MC–Cu (%)	
Samples	Wt%	Atom percent (%)	Wt%	Atom percent (%)	Wt%	Atom percent (%)
C	54.09	61.70	52.41	59.76	49.61	59.56
O	44.83	37.88	46.68	39.96	43.00	38.76
M	0.71	0.18	0.63	0.16	7.39	1.68

3.4. Thermal degradation of the cellulose-supported complexes

In order to investigate the thermal stability of the cellulose-supported complexes, thermogravimetric and derivative thermogravimetry analyses (TG and DTG) were measured and shown in Figs. 7 and 8.

Fig. 7 shows that the onset of degradation for control cellulose was at 344.1℃. The onsets of degradation for MC–Co, MC–Mn, and MC–Cu, were at 328.2, 332.3, and 285.2℃, respectively. Compared with the control cellulose, the onsets of degradation for MC–Co, MC–Mn slightly decreased. The onset degradation temperature of MC–Cu significantly decreased to 285.2℃. It can be attributed that the much more copper ions on the surface of the cellulose-supported complexes might catalyze cellulose pyrolytic degradation. The mass loss yields of the residue for MC–Co, MC–Mn, and MC–Cu were 17.10, 15.91, and 18.45% at 600℃ in nitrogen, respectively. It is obvious that the mass loss yields of the residues of the three cellulose-supported complexes were higher than that of the control cellulose (5.32%).

Fig. 8 indicates that the maximum temperatures of mass loss for MC–Co, MC–Mn, and MC–Cu, were at 353.1, 356.4, and 332.8℃, respectively. The temperatures for MC–Co and MC–Mn were basically in agreement with that for the control cellulose, which was 354.6℃. The maximum rates of mass loss for MC–Co, MC–Mn, and MC–Cu were 17.84, 19.68, and 8.76%/min, respectively. It is assigned to the typical thermal degradation of cellulose. However, because of the effect of the metal complexes, the maximum rates of mass loss for three materials were slower than that of the control cellulose (21.75%/min).

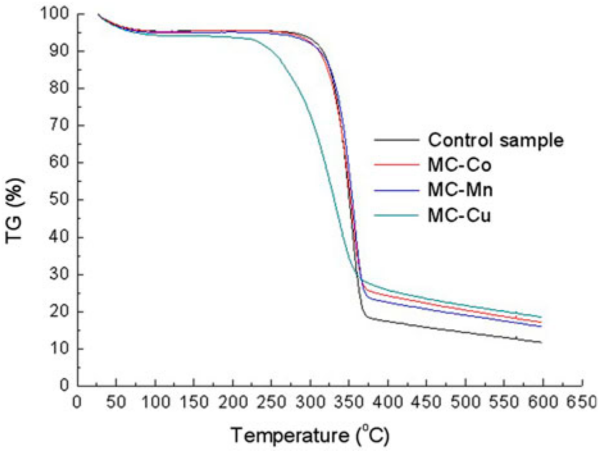

Fig. 7. TG curves of the control sample and cellulose supported complexes.

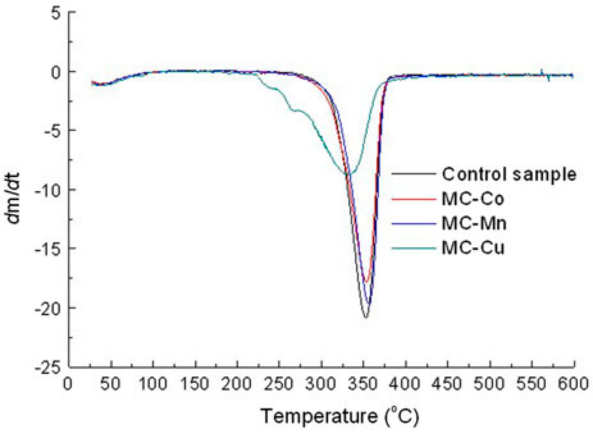

Fig. 8. DTG curves of control sample and the cellulose supported complexes.

3.5. Catalytic property of the cellulose-supported complexes for H_2O_2 decomposition

The catalytic property of the cellulose-supported complexes for H_2O_2 decomposition was measured. The catalytic reaction of H_2O_2 was carried out at 40℃. The concentration of the residual H_2O_2 was measured by means of the titration with 0.02 mol/l $KMnO_4$. The results of catalytic decomposition of H_2O_2 are shown in Fig. 9.

It was found that the control sample had not obvious catalytic activity for H_2O_2 decomposition. The modification cellulose with 3,5-bis {2-hydroxyphenyl-5-[(2-sulfate-4-sulfatoethylsulfonyl-azobenzol) methylene

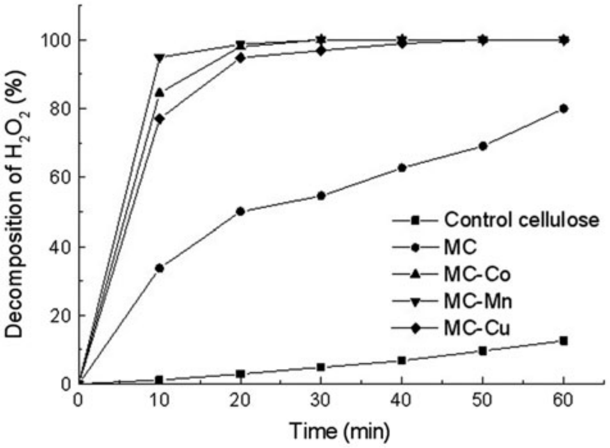

Fig. 9. Catalytic activity of the cellulose supported complexes for H_2O_2 decomposition.

amino]} benzoic acid azo-Schiff base (MC) had the noticeable catalysis activity. The rate of H_2O_2 decomposition with MC reached 50% in 30 min. The three cellulose-supported complexes, MC–Co, MC–Mn, and MC–Cu had strong catalytic activity for H_2O_2 decomposition. H_2O_2 decomposition rates in 10 min were 84.54% for MC–Co, 94.87% for MC–Mn, and 77.21% for MC–Cu, respectively. H_2O_2 were completely decomposed in 30 min with MC–Co, MC–Mn, or MC–Cu. Manganese complex, MC–Mn, had the strongest catalytic activity among the three complexes. The order of catalytic ability of them for H_2O_2 decomposition was MC–Mn > MC–Co > MC–Cu.

4. Conclusions

Cellulose fiber surface was modified with 3,5-bis {2-hydroxyphenyl-5-[(2-sulfate-4-sulfatoethylsulfonyl-azobenzol) methylene amino]} benzoic acid. Grafting reactive rate on cellulose was 24%. The grafting quantity of BHSABA Schiff base on cellulose fabric was 5.08×10^{-2} mmol/g. The modified cellulose surface had the strong absorbance at 350–500 nm. The assembly quantity of transition metal ion on cellulose surface was Co content 0.57% for MC–Co, Mn content 0.61% for MC–Mn, and Cu content 1.53% for MC–Co, respectively. The elemental analyses of the metal cellulose-supported complexes, MC–Co, MC–Mn, were well agreed with the proposed composition. Cu content of MC–Cu was higher than that of the theoretical calculation. The morphological structure of the cellulose-supported complexes had no obvious difference. Their appearances were smooth. The onsets of degradation for MC–Co, MC–Mn, slightly decreased. The onset degradation temperature of MC–Cu obviously decreased. The three cellulose-supported complexes had strong catalytic activity for H_2O_2 decomposition. The order of catalytic ability of them for H_2O_2 decomposition was MC–Mn > MC–Co > MC–Cu.

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